

Adhesion and Silicone Adhesives



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Application Note

Adhesion

The term “adhesion” refers to the state in which two substrate surfaces are held together by interfacial forces, or bonds¹. Adhesion can come about in different ways and last for varying amounts of time depending on such factors as substrate surface preparation, adhesive used, and compatibility of the substrates being held together by the adhesive. In some cases, failure of the adhesive bond results in catastrophic device failure; in other circumstances, a strong adhesive bond is not desirable. Generally, the stronger the bond between substrate and adhesive, the longer adhesion will last. Tacky gels and pressure-sensitive adhesives have been used for a variety of applications in which temporary bonds are needed, such as in transdermal patches and processing aids².

Most silicone adhesives are made of silica-filled silicone polymers that cure in place by crosslinking. The overall mechanical strength is known as cohesive strength. The strongest bonds are those which occur between atoms from chemical bonding; however, adhesion is not limited to chemical bonding. Conversely, materials can have the ability to form a strong “bond” when brought into contact with another surface for a short time (seconds) under light pressure. This short-term, pressure-induced attachment is known as tack². Tack and adhesion are influenced by how a force (and how much of it) is applied to the bond, as well as the bond thickness (thinner can be better)¹.

Basic Adhesion Mechanisms

Many factors affecting adhesion can be manipulated according to an adhesive’s intended use, but the following compilation entails fundamental concepts essential to how silicone adhesives function from a chemical and mechanical perspective:

Chemical Bonding

- Chemisorption - Covalent bonding
 - Bond formed between adhesive and substrate when their molecules share electrons
 - Strongest type of bonding
- Adsorption - Hydrogen bonding, Van der Waals, polar interactions
 - Bonding between hydrogen and unpaired electrons
 - Not as strong as covalent

Mechanical Adhesion

- Mechanical interlocking of pieces
 - Dependent on strength of materials, not chemistry at the surface
 - “Lock and Key”

Interdiffusion

- Occurs when materials are soluble in each other

- Occurs with polymers and alloys
- Similar to mechanical interlocking on molecular level

Evaluating Adhesion

As regards evaluating an adhesive, failure modes can be categorized in two different ways: adhesive failure and cohesive failure. **Adhesive failure** occurs when the bond fails between adhesive and substrate.

Cohesive failure occurs when the bond fails through the adhesive/silicone, leaving a coating of silicone on both of the substrates (test panels). This means the adhesive bond is stronger than the silicone. (For information on adhesion test methods, see Appendix.)



Figure 1: Adhesive and cohesive failure.

Substrate Considerations

Adhesive performance is largely dependant upon the substrate to which it is applied¹. How receptive a substrate is to an adhesive is generally related to wettability, which refers broadly to substrate acceptance of a coating. Poor wetting is indicated by beading, which means the coating is not flowing, or “wetting out”³. Beading is illustrated in Figure 2 below.

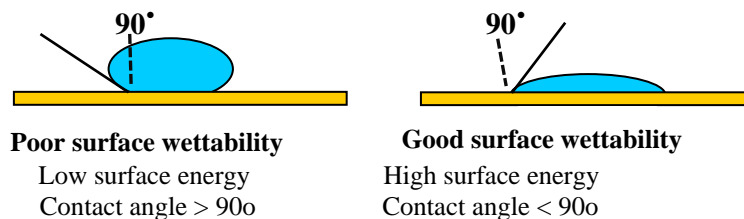


Figure 2: Comparison of poor surface wettability and good surface wettability.

Poor wetting will result in poor adhesion, and good wetting *may* result in good adhesion. If a substrate has low surface energy, or more of a resistance to an adhesive, one should take steps to make the surface more reactive before applying the adhesive. The following factors⁴ influence how “welcoming” a substrate will be to an adhesive:

- Surface area
 - Rough surface creates more area for chemical and mechanical bonding
 - It is more difficult to bond to polished and smooth surfaces.
- Surface cleanliness
 - Eliminates surface contaminants (cutting oils, etc.)
 - Removes moisture
- Surface chemistry-
 - Molecular compatibility with substrate—wettability
 - Chemical reactivity with adhesive

Methods of Improving Adhesion

Choosing an adhesive should be based on its molecular compatibility with the substrate to which it is being applied. Improving adhesion to a substrate can be accomplished by modifying the surface of the substrate to increase the reactivity and compatibility of the substrate, and/or by adding adhesion promoters to the silicone formulation itself. Each of these methods has trade offs but can dramatically increase the bond

strength if engineered well. Surface preparation of the substrate is often crucial. Methods of surface modification include the following³:

1.) Primer

- Applied to surface as a preparatory step
- Ideally forms monolayer on substrate
- Modifies substrate surface for better wetting of silicone
- Covalent bonds across joint when cure complete

2.) Flame/plasma/corona treatment

- Cleaves bonds to expose reactive groups
- Surface is more reactive with adhesive.

Primers

Silane primers are used to promote adhesion between two non-bonding surfaces. Prior to applying the adhesive, a monolayer of primer can be applied to a substrate as surface preparation for the adhesive. The silane in primer is a coupling agent containing two reactive groups, one which reacts to the substrate and another which reacts to the adhesive. By increasing the covalent bonds between the adhesive and substrate, the amount of force needed to break adhesion is increased and higher lap shear values are achieved. Primers also greatly increase the silicone adhesive's ability to wet out the substrates. (See "Choosing a Silicone Primer Adhesive System" and "Application and Storage Recommendations for NuSil's Primers" for more information on primers.) Figures 3 and 4 depict the process of interaction between silanols in the primer and the substrate to which the primer is being applied. Figure 3 is the first step of the primer reaction, during which the silane undergoes hydrolysis into the more reactive silane.

Figure 3: Step 1. In the ideal silane primer reaction, the silane is hydrolyzed. (NuSil Recommends 30 minutes minimum at 30-70% RH).

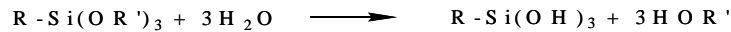


Figure 4 is the second step, during which the reactive silanols react onto alcohol functional molecules that will form covalent bonds with the primer and the substrate.

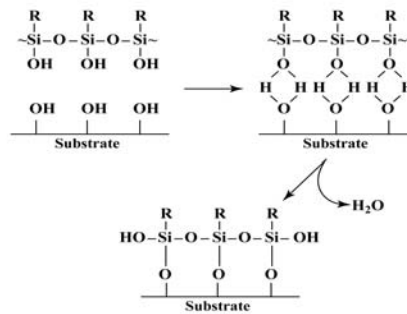


Figure 4: Step 2. After silane is hydrolyzed, silanols react with the substrate.

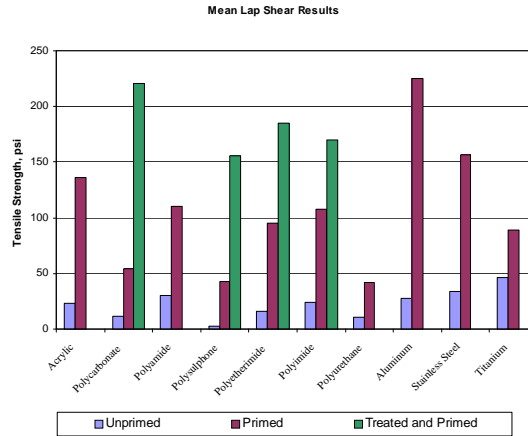


Figure 5: Lap shear results on primed, unprimed, and treated and primed substrates.

Flame/Plasma/Corona Treatment

Applying flame, plasma or corona to substrates before applying a silicone adhesive, makes the substrate surface more reactive to the silicone⁵. These treatments may accomplish the following:

- Oxidize surface which increases reactivity to adhesive and/or primer
- Increase surface energy
- Hydrophobic => Hydrophilic

Flame and corona treatments each generate radical oxygen atoms that oxidize the substrate's surface. From gases such as oxygen, plasma treatment creates high energy molecules that form oxygen-containing reactive groups on the substrate, initiating reaction with the adhesive. The best method of treatment depends on the substrate composition, topography and surface area. Because substrate response generated by flame, plasma and corona decreases over time, it is recommended to apply the adhesive within one week of treatment for the best performance.

Adhesion Promoters

Adhesion promoters play the same role that primers do. Unlike primers, adhesion promoters are coupling agents added to the Part A or Part B by the manufacturer. In other words, rather than existing independently of the silicone, they are mixed in as part of the silicone adhesive.

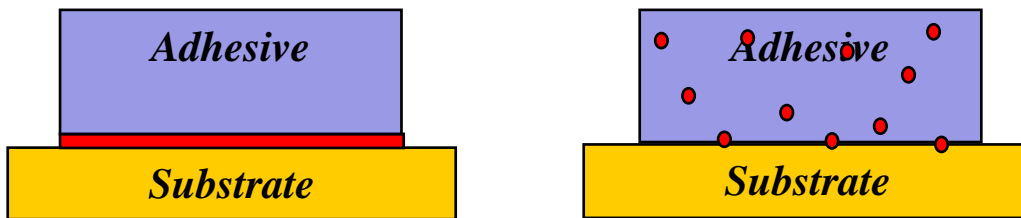


Figure 6: Pictorial comparison of substrate and adhesive with primer (left), and substrate and adhesive with adhesion promoters (right).

Adhesion promoters eliminate the extra step of adding a primer, but with them the silicone takes longer to cure. Sufficient time must be allotted for the adhesion promoters to react with the substrate before the adhesive cures. For processing capabilities and application requirements, one must consider that adhesion promoters may affect the silicone's rheology, changing aspects such as shelf life or transparency.

Silicone Adhesives

While various types of materials can be used as adhesives, silicone is consistently chosen for its many inherent properties and overall versatility. Although silicones are most commonly known for their

resistance to moisture (valuable for projects such as bathroom caulking) they are also used in applications requiring their proven biocompatibility and ability to withstand harsh thermal conditions. Particular to the medical field, acetoxy-cured silicone adhesives can be engineered to adhere well to other substrates used in medical devices, such as polyurethanes and titanium. Very relevant to the electronics, aircraft and aerospace industries, silicone adhesives can remain elastic below 0 °C (T_g @ -120 °C) and resist breakdown at temperatures > 200 °C. In addition, their low modulus absorbs stresses during thermal cycling when adhering substrates with different Coefficients of Thermal Expansion (CTE). This prevents warpage, delamination and substrate failure.

Types of Silicone Adhesives

Silicone adhesives typically use either an addition or a condensation-cure system. Addition cure adhesives cure evenly and equally throughout by way of a catalyst, namely platinum. Condensation cure systems require moisture for the crosslinking reaction and are available with different crosslinkers and typically use a tin or titanate catalyst. Because these adhesives require atmospheric moisture to cure, heat acceleration of the cure is limited and vulcanization occurs from the outside in. Based on their different properties, addition cure adhesives and condensation cure adhesives work well in certain environments and applications, and not so well in others⁸.

Addition Cure Adhesives

Addition-cure silicones have been used in many applications for more than 30 years. They have a proven history in medical applications as well as in harsh environments. The formulations using platinum catalyzed addition cure chemistries are very versatile where the cure rate, mechanical properties and flow characteristics have almost an endless range of options. Historically, compounds known as “inhibitors” have been used to control the cure rate and pot life. Addition-cure silicones can be designed to be applied on large platforms and cure at room temperature, or for dispensing on automated equipment where the pot life needs to be relatively stable to prevent the drop size decreasing under constant pressure. The inhibitor chosen can affect the minimum cure temperature, so contact NuSil for questions on adjustable cure rates.

It should be noted that certain undesired inhibitors in the environment, such as amines or sulphur, may unintentionally come into contact with silicones, especially during manufacturing. Such materials are referred to as “poisons” because they cause unwanted inhibition that is out of the manufacturer’s control. These can temporarily and sometimes permanently halt or slow the vulcanization of addition cure adhesives by isolating any platinum contained in the material. Because platinum poisons may be contained in the substrate, it may be beneficial or even necessary to heat the substrate before applying an adhesive in order to bake out these poisons.

General Properties for Addition-Cure Adhesives

- Excellent when adhering large platforms
- Can be formulated for room temperature cure as well as heat cure, for reasons such as a longer pot life
- Can be heat accelerated
- Minimal shrinkage — no cure reaction byproducts
- Designed cross linking with a platinum catalyst allows these silicones to be formulated to provide a wide range of physical properties, adjustable work times and reaction rates due to inhibition, and manageable mix ratios such as 1:1, 10:1, 20:1.

Condensation-Cure Adhesives

Condensation-cure adhesives, as stated earlier, generally use either acetoxy, oxime or alkoxy crosslinkers. All of these follow the same curing principle of condensation, meaning they need to be exposed to atmospheric moisture and will have 5% to 7% weight loss during cure due to leaving groups evaporating. The evaporation rate varies based on the type of crosslinker used in the system and results in dimensional shrinkage. All condensation-cure adhesives offer excellent adhesion to metals and a variety of other substrates; however, they have distinct attributes that dictate use of one type over the other⁸.

Acetoxy has an acidic acid curing by-product and can be used in such applications as coating large areas, e.g. aircraft wings, with dispersions, or for use in such medical devices as pacemakers and catheters. Most

acetoxy products have a cure time of 72 hours, and tack-free time is typically seven to 10 minutes. Adhesion to metals is also increased with acetoxy adhesives due to etching from the acidic leaving group. This creates strong adhesion between silicone and the substrate, yet (1) acetic acid can corrode electronics and (2) the leaving group causes mass loss and shrinkage. Since the leaving group smells like vinegar, one can use the presence or lack of this scent to determine whether the material has fully cured.

Oxime, unlike acetoxy, can be used in electronics. It can also be used in sealants for extra-terrestrial applications when the material is processed properly. Oxime materials generally have a cure time of seven days and, often, tack free times range from 20 to 30 minutes. As with acetoxy, the leaving group causes mass loss and shrinkage; however, with oxime, the leaving group has lower volatility, resulting in fewer voids in the adhesive coating. Oxime is slightly corrosive and has less adhesive strength than the acetoxy cure.

Alkoxy-cure adhesives are typically two parts and are employed when platinum cannot be used due to unwanted inhibition, or because it is a legacy material. These have non-corrosive alcohols as leaving groups and were traditionally used in electronics applications. The one-part versions can have limited shelf life stability.

General Properties for Condensation-Cure Adhesives

- Use is heavily dependent on exposed surface area and environmental conditions (%RH & temperature).
- Most are liquid with manageable viscosities.
- Tin catalyst avoids corrosion and inhibition.
- Longer cure times (usually 3-10 days)
- Challenging mix ratios such as 100:0.5, 100:3.8
- Shrinkage due to volatile leaving group

Table 1: Property summary of the main crosslinkers used in condensation cure silicone adhesives

Cure Type	General Characteristics	Properties of Leaving Group
<p>Acetoxy</p> <chem>—O—C(=O)—CH3</chem>	<ul style="list-style-type: none"> • One-Part • Corrosive, acidic. Etches surfaces • Commonly used as adhesives and sealants • Very fast curing • Depth of cure limited • Catalyst typically a tin carboxylate 	<p>Acetic Acid</p> <ul style="list-style-type: none"> • MW: 60.05 g/mol • BP: 118° C • VP: 10.5 mmHg • XL/oc: Methyl or Ethyl triacetoxysilane • % Mass Loss: >65-75%
<p>Alkoxy</p> <chem>—O—R</chem>	<ul style="list-style-type: none"> • Non-corrosive, alcohol leaving group is a weak acid • Two-Part -Catalyst is typically a tin carboxylate -Can be used for thicker sections and faster cures • One-Part -Catalyst is typically a titanium alkoxide 	<p>Ethanol</p> <ul style="list-style-type: none"> • MW: 46.08 g/mol • BP: 78.3° C • VP: 44 mmHg • XL/oc: Ethyl orthosilicate (EOS) • % Mass Loss: >71%
<p>Oxime</p> <chem>—O—N—C(R)2</chem>	<ul style="list-style-type: none"> • One-Part • Slightly corrosive, basic. • Lower volatility of leaving group results in less voids • Often used as silicone inks and coatings • Catalyst is typically a tin carboxylate 	<p>Methyl Ethyl Ketone</p> <ul style="list-style-type: none"> • MW: 58.14 g/mol • BP: 153° C • VP: 4 mm Hg @ 150° C • XL/oc: Methyl trimethoxysilane (MOS) • % Mass Loss: >78%

Adhesive Form Factors

Silicone adhesives can be delivered in a variety of form factors to aid in increased throughput and mechanical performance. This means that from one silicone to the next, properties such as cure chemistry, cure temperature, and catalyst may vary. When choosing which adhesive to use, these and other properties, such as strength or temperature resistance, should be considered based on intended application.

Adhesives can be made available in the following form factors:

- One Part – Liquid for cure in place
 - RTVs (room temperature vulcanization)
 - Platinum-catalyzed (frozen until time of use)

- Two-part adhesives – Liquid for cure in place
- Film adhesives
- PSAs (pressure-sensitive adhesives)

One-Part Adhesives

One-part adhesives are available in a liquid form uncured where the viscosity can be optimized to have high flow to coat complex geometries, or low flow if applied to a vertical surface or to control migration of the adhesive prior to cure.

One-part adhesives, also known as condensation-cure adhesives, are typically room temperature vulcanizing and use either acetoxy, oxime, or alkoxy crosslinkers. The main advantages of using a one-part RTV are that the adhesive can be immediately applied to the substrate and heat does not need to be added to induce curing. However, because the cure is temperature and humidity dependant, the depth of cure is limited. Because they also lack platinum, one-part RTV adhesives are not as susceptible to inhibition and are thus ideal for applications wherein platinum inhibition is a concern.

One-part, platinum-catalyzed adhesives, such as glob tops and die attach, are becoming more popular, especially for electronics applications. This is due to the use of addition-cure chemistry: These adhesives are non-corrosive, have minimal shrinkage, and the cure can be heat accelerated for faster throughput. The logistics of shipping and handling frozen materials should be considered, and extra attention should be paid to this detail if using a one-part, addition-cure adhesive.

Two-Part Adhesives

Two-part adhesives are mainly available in addition-cure chemistry but can be formulated using oxime or alkoxy condensation cure. These are also liquid when uncured and are available in a variety of viscosities and cure rates for a multitude of applications. They can easily be packaged into dual cartridge kits to be dispensed directly onto the substrate and to eliminate the added step of mixing and de-airing. They are also recommended when adhering large panels or when curing in closed systems.

Condensation-cure adhesives with a tin catalyst are ideal for large surface areas that cannot be heat cured. An oxime cure silicone has been used historically to bond solar cells to polyimide for space-grade solar modules. Addition cure silicones can also be designed to be used in over-molding and insert-molding, as opposed to adhering a silicone part onto a substrate in a separate process.

Film Adhesives

Film adhesives can be an alternative to one part or two part liquid adhesives. They are uncured films that have high “green strength” and do not flow in the uncured state. They can be made in various thicknesses based on the bond line thickness needed, and the silicones’ high cohesive strength is very robust under thermal cycling. The films are initially uncured and a platinum-containing “activator” is applied to the substrates similar to the way a primer is applied. Once the “activator” has remained on the substrates for at least 30 minutes at 30%-70% relative humidity, the film can be applied—bubbles removed with vacuum and/or pressure—and then cured with heat or ambient conditions.

Film adhesives do not flow and so provide a controlled bond line thickness. They work well between at least one flexible substrate and another substrate, such as between solar cells and backing sheets, as well as on flexible circuit boards. They can also be used as die cut pieces for specific patterns and shapes. On the other hand, they cannot fill voids or seep into corners of small parts, and it is difficult to apply a film adhesive on odd shapes and to reposition one once it has been applied. Film adhesives are available in space grade and electrically conductive versions as well.

Pressure-Sensitive Adhesives and Tacky Gels

The examples above are designed to have excellent covalent bonding and high cohesive strength, which are not always desirable. Silicone pressure-sensitive adhesives (PSAs) and tacky gels have excellent wetting to a variety of surfaces and are used for making tapes and films. Due to the tackiness of the PSA or cured gel, adhesion occurs when the film contacts another surface for a short time under light pressure. On average PSAs have an adhesion strength of up to 2 or 3 lbs/in (.91-1.36 kgs/cm) because they attach to substrates by

a relatively weak form of adhesion—adsorption. The film can be removed and the strength needed to remove the film will depend on contact wetting, interdiffusion/adsorption, and cohesive strength of the film material.

Silicone PSAs and tacky gels are extremely versatile: They have traditionally been used for transdermal patches and wound care, and can also be formulated to be space grade. PSAs can be delivered in solvent for the end user to make the tape on their preferred release liner and backing sheet. Tacky gels can also be cured into the final form factor by the end user and reinforced with a scrim if additional cohesive strength is needed. Once these are made into a film, they can also be die or laser cut into the desired shapes.

PSA and tacky gels are excellent choices where the bond needed is temporary and minimal to no residue is desired. These can be used for transdermal patches in drug delivery applications, for temporary bonding when transferring parts to other processes, for applications where re-work is likely to be needed, or for dust collectors. Space-grade PSAs can also be designed to maintain adhesion at elevated temperatures. Tapes and films are the future direction for many bonding applications due to the versatility in formulations in silicone, release liners, scrims, and release liners².

Summary

Not only are there many different types of adhesives, there are also several types of silicone adhesives. Your choice of a silicone adhesive—whether a one part RTV adhesive, a two part adhesive, a film adhesive or a pressure sensitive adhesive—should depend on the substrates you are using and the application(s) you are undertaking. While silicone adheres well to a variety of substrates on its own, primers and other preparations do help improve adhesion. Because finding the most suitable adhesive and/or primer can be difficult, NuSil offers custom formulation of primers and adhesives as needed. Especially when communicating unique requirements, having a good relationship with your material supplier can be helpful.

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Appendix

Test Methods

NuSil performs the following tests on its adhesives. Equivalent ASTM numbers are given if applicable.

Tests for Covalent Bonding

NuSil Test Number	Test Name	Comments	ASTMS Number
TM039	Adhesion	Qualitative	D3359
TM010	Lap Shear	N/A	D1002
TM087	Silicone Peel Strength	Tests adhesives to silicone	D1876
TM076	Peel Strength of Cured Bi-layers from Silicone Dispersions	N/A	N/A

Tack and Cohesive Strength (PSAs and Tacky Gels)

NuSil Test Number	Test Name	Comments	ASTM Number
TM122	PSA Blunt Probe Tack	N/A	D2979
TM085	Release Force of PSA	N/A	D766
TM152	PSA Static Shear	Maintain adhesion at 175 C with 50-gram weight attached	N/A
TM103	Surface Tack	Mainly used on gels; Test adhesion to aluminum	D429

Adhesion Marketing Literature

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Fast-Cure Silicone Adhesives for Medical Device Applications:

<http://www.nusil.com/library/papers/Fast-Cure%20Silicone%20Adhesives%20for%20Medical%20Device%20Applications.pdf> April 2009

Tacky Gels for Healthcare Applications:

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Choosing a Silicone Primer/Adhesive System (Healthcare Applications):

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